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Modeling of the Dissolution Kinetics of Arbutus Wild Berries-Based Tablets as Evaluated by Electric Conductivity

Permodelan Pembubaran Kinetik Arbutus Beri Liar Berasaskan Tablet Dinilai oleh Kekonduksian Elektrik

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ABSTRACT

*Lyophilized powder (LP) from Algerian arbutus wild berries (*Arbutus unedo *L.) was obtained. This present paper reports about the dissolution (releasing) properties of LP-based tablets, evaluated through the electric conductivity (EC) of distilled water which is employed as surrounding medium, at three different temperatures (291, 298 and 309 K). In addition to this, secondary physicochemical characteristics such as elementary analysis, color and compressibility were evaluated. Regarding the modeling of ionic transfer, among the three tested models, namely Peleg, Singh et al. and Singh and Kulshestha, the latter seems to be the most appropriate (R^2 = 0.99), particularly in the case of compacted tablets under 2000 Pa. The temperature dependence of the dissolution process was also studied applying Arrhenius equation (R^2>0.8) which allowed to deduce the activation energy, ranging from 18.7 to 21.4 kJ.mol^{-1} according to the model and compression force employed.*

Keywords: Arbutus wild berries (Arbutus unedo *L.*); *dissolution; electric conductivity; powder; tablet*

ABSTRAK

*Serbuk lyophilized (LP) dari Arbutus beri liar Algeria (*Arbutus unedo *L.) telah diperoleh. Kertas ini melaporkan tentang sifat pembubaran (pelepasan) tablet berasaskan LP, dinilai melalui kekonduksian elektrik (EC) air suling yang digunakan sebagai medium kelilling, pada 3 suhu yang berbeza (291, 298 dan 309 K). Di samping itu, ciri fizikokimia sekunder seperti analisis asas, warna dan kadar mampatan telah dinilai. Mengenai model pemindahan ionik, antara tiga model yang diuji ialah Peleg, Singh et al. dan Singh dan Kulshestha, dengan yang terakhir paling sesuai (R^2 = 0,99), terutamanya dalam kes tablet yang dipadatkan di bawah 2000 Pa. Pergantungan suhu dalam proses pembubaran juga dikaji menggunakan persamaan Arrhenius (R^2> 0.8) yang dibenarkan untuk menyimpulkan tenaga pengaktifan, antara 18.7 hingga 21.4 kJ.mol^{-1} mengikut model dan mampatan daya yang digunakan.*

*Kata kunci: Arbutus beri liar (*Arbutus unedo *L.*); *kekonduksian elektrik; pembubaran; serbuk; tablet*

INTRODUCTION

Arbutus wild berries (*Arbutus unedo* L.) are Mediterranean typical fruits generally not consumed in fresh form (Ayaz et al. 2000) but after processing (Pawlowska et al. 2006). Like other plants which are endowed with wonderful defense system assured by various biopharmaceuticals (Rahman 2007), the berries are also known to be used in folk medicine as antiseptic, diuretic and laxative (Pallauf et al. 2008). Moreover, this wild fruit is rich in numerous nutriments like calcium and potassium (Ozcan & Hiciseferogullari 2007), antioxidants (Tawaha et al. 2007) and sugars with a content of about 0.47 kg.kg^{-1} $_{\text{dry basis (db)}}$ (Alarco E Silva et al. 2001). Among 27 various investigated Algerian fruits, Allane and Benamara (2010) have demonstrated that arbutus berries present the highest reducing power (~ 20 mg vitamin C equivalent.g^{-1} whole fruit), Ruiz-Rodríguez et al. (2011) earlier supported that the higher antioxidant potential of the arbutus berries may be due to the activity of various bioactive components including vitamin C. Based on the fact that any herbal or botanical material containing vitamins and minerals are considered as a dietary ingredients (Devla et al. 2011), arbutus berries may be classified as a dietary supplement.

Despite the importance in the engineering field of powdered food products, the scientific investigation of the latter remains insufficient (Murieta-Pazos et al. 2012). In particular, the powders from arbutus berries (*Arbutus unedo* L.), to the best of our knowledge, never been studied. But, we have recently evocated in a brief communication the opportunity to produce tablets from arbutus berries powder (Benamara et al. 2012).

This present paper reports essentially about the dissolution (releasing) properties of LP-based tablets, evaluated through the electric conductivity (EC). Fruit tablets are of strong interest for various reasons like extended shelf life and functional properties. Unfortunately, as what was mentioned before, few works are found to be devoted to similar tablets from whole fruits: date (*Phoenix dactylifera* L.) (Adiba et al. 2011), *Terminalia chebula* fruit (Prakash et al. 2011), baobab pulp fruit powder as hydrophilic excipient for pharmaceutical tablets (Arama et al. 1989).

Material and Methods

Fruit and fruit powder

Fully ripe Arbutus berries (Figure 1(a)) were randomly picked at various trees in Kabylian region (northern Algeria) in winter 2011. The fruit is submitted to freeze drying at 109 K (4.5 Pa) during 2 days using lyophilizer of type 'Christ Alpha 1-4LD' provided with vacuum pump (RZ 6, max pressure = 0.04 Pa). The dried product is then ground and sieved (sieve of type Euromatest-Sintoo, NFX11-501) to obtain homogeneous powder (LP) (Figure 1(b)) which is finally kept in closed glass flask at 277 K.

Dissolution test

First, the LP was characterized by some physicochemical criteria, including pH, water content, °Brix, ash, color parameters in CIELab system (Hunter-Lab tristimulus colorimeter Thermocontrol Sarthorieus), flow rate (g/s) (Patil et al. 2012), Carr's index (CI, %) (characterizing the compressibility) (Olayemi et al. 2011) and Hausner's ratio (HR) (characterizing the flowness) (Olayemi et al. 2011). Hydraulic laboratory press was employed to obtain tablets from LP by direct compression, applying two pressures (1000 and 2000 Pa). It is generally recognized that among the three tabletting methods applied, namely wet granulation, dry granulation and direct compression, the latter is the most preferred because of its numerous advantages, especially its simplicity (Patel & Bhavsar 2009).

Subsequently, the dissolution- related properties of tablets at different temperatures 291, 298 and 309 K were investigated by studying the variation versus time of the electric conductivity (EC) of the dissolution (surrounding) liquids simulating physiologic media (only results related to bidistilled water are presented here) according to the simple system proposed by Jayjock et al. (2005), knowing that the dissolution tests are complex (Yu 2008). In fact, while the common standard dissolution testing like described by United States Pharmacopoeia is generally used to investigate solid delivery systems, some authors have nevertheless adapted a specific methodology. Thus, taking into account the limitation of performing the USP dissolution test in relation to their specific conditions, Young et al. (2008) have assembled a large reservoir upon a magnetic stirrer to simulate the pharmacopoeia dissolution test. The electrical conductivity was measured by means of conductivity meter (Type EC 214 HANNA).

Frenning et al. (2002) have already used the electric conductivity measurement to explore drug dissolution of tablets containing micronized cellulose and NaCl as a model drug, whereas Brielles et al. (2008) and Chantraine et al. (2006) have employed the electric conductivity to study the phenomenon of dissolution of detergent tablets. On the other hand, Mikac et al. (2007) monitored the dissolution of tablets containing NaCl and carboxylic acids by following the conductivity changes in surrounding medium by means of electric current density imaging technique, whereas Gauza and Kubisz (2010) performed tablets from collagen in view to study the water release process of this protein, adopting the electric conductivity as quantification criterion of water transfer.

Three mathematical models were used to describe the dissolution of tablets from arbutus-berry powder. The use of three models related to the masse transfer, is dictated principally by the principle of parsimony (Hill 2006). Some types of such models are adapted to study the release process. Weibull model, widely used in food processing, is for example adapted to investigate dissolution/release process (Vudathala & Rogers 1992) and indicates the accumulated fraction of the drug in solution at any time (Costa & Sousa Lobo 2001). This is why we attempt to apply analogue models for describing the dissolution process evaluated through the EC of the surrounding media. The dimensionless form of the response seems to us to be particularly interesting to investigate. The three models on which we based our research are detailed as follows.

-Peleg model (Peleg 1988), is widely used to study, before all the rehydration processes, but, Boussetta et al. (2009) have applied this model to investigate the extraction process of polyphenols from grape pomace (*Vitis vinifera* L.).

The Peleg's model as adapted to the present study can be written as follows:

$$\sigma = \sigma_0 + t / (k_1 + k_2 t),$$

where σ and σ_0 are the initial and at any time (t) conductivity ($S.m^{-1}$), respectively; t is the dissolution duration (s); k_1 is the Peleg's rate constant ($m.s.S^{-1}$) and k_2 is the Peleg's capacity constant ($m.S^{-1}$).

- Model of Singh and Kulshrestha (Singh & Kulshrestha 1987):

$$(\sigma_e - \sigma) / (\sigma_e - \sigma_0) = 1/(kt + 1),$$

where s_e is the equilibrium conductivity and k is the rate constant (s^{-1}).

- Model of Singh et al. (Singh et al. 1981):

$$(\sigma_e - \sigma) / (\sigma_e - \sigma_0) = \exp(-kt),$$

where k is the Singh et al.'s constant (s^{-1}).

To elucidate the temperature dependence of the model constants, the following well known Arrhenius equation was used:

$$k = k_o \exp(-E_a/RT),$$

where k_o is the pre-exponential factor, E_a is the activation energy ($kJ.mol^{-1}$), R is the constant ($8.134\ J.mol^{-1}\ K^{-1}$) and

T is the absolute temperature (K). By plot the regression right ln k versus 1/T it becomes possible to deduce Ea.

Statistical Analysis

The statistical analysis of experimental data was performed using Origin software version 7.5.

Goodness of fit of the selected models was evaluated by means of the coefficient of determination (R^2), sum of square error (SSE) and root mean square error (RMSE).

$$SSE = 1/N \sum_{i=1}^{N} \left(X_{exp} - X_{cal}\right)^2.$$

$$RMSE = \left[1/N \sum_{i=1}^{N} \left(X_{cal} - X_{exp}\right)^2\right]^{1/2},$$

where X_{exp} is the experimental value, X_{cal} is the value predicted by the model and N is the number of experimental measurements.

Results and Discussion

Round tablets of (400±1) mg weight, 12 mm diameter and 2 mm thickness were obtained from LP by direct compression, applying two different compression forces (Figure 1(c)). Some physicochemical properties of the LP (Figure 1(b)) are given in Table 1.

Taking into account the importance of the external aspect as quality criterion, the color parameters (L*, a* and b*) of the LP are similar to those (L* ~ 30, a* ~ 10 and b* ~ 25) reported by Orak et al. (2011) for fresh strawberry tree fruits from Turkey, which confirms the efficiency of freeze drying concerning the intrinsic color preservation (Kampuse & Volkova 2009).

Moreover, the applicability of the direct compression for arbutus berry fruits can be easily observed considering the fact that two following conditions are met (Göczo et al. 2000): Powder flowability with HR<1.25 (see the added Table 1), limit value recognized by Panda et al. (2008); and its compactability/compressibility with CI value <15 (see the added Table 1), as required by the European pharmacopea. Also, Arbutus berry fruit as a rich pectin-material (Ruiz-Rodríguez et al. 2011) can be presumed to be able to undergo direct compression, Salbu (2011) demonstrates that certain pectin present an interesting potential as direct compression excipients in tablets.

(a) (b) (c)

FIGURE 1. Photographs of the arbutus berry whole fruits (a), their freeze-dried powder (b) and tablets (c) obtained under pressure of 1000 (left) and 2000 Pascal (right)

TABLE 1. Some physical properties of powder from freeze-dried strawberry fruits

Parameter	Value
pH	3.83±0.08
Water content (%)	2.8
°Brix (%)	90.00
Ash (%)	3.01±0.03
L*	64.48
a*	8.51
b*	24.11
h°	70.56
C*	25.56
CI (%)	13.69±0.26
HR	1.15±0.00
θ° (Angle of repose)	35.00
Flow rate (g/s)	10.00

Date are represented as mean ± SD (*n*=3), HR = Hausner's ratio; CI = Carr's index, h° = Hue angle = arctan (b*/ a*) and C* = total color = $(a^{*2} + b^{*2})^{0.5}$

These tablets can be used as such and/or as carrier for natural active ingredients from plants. As supported by Madrigal-Carballo et al. (2010), the challenge now consists of the incorporation of natural bioactive molecules into dosage forms for preventive and therapeutic applications, knowing that the use of natural materials as drug delivery vehicles arouses interest among scientists, due to numerous advantages like their accessibility (Amin et al. 2012). This is why it seemed useful to investigate the behavior of tablets in different surrounding liquids simulating physiologic media, choosing the EC as indicator of release properties of arbutus berry tablets (only data for distilled water are shown here). Obviously, the transport phenomenon concerns electric charge carriers, including organic and non-organic ions. It must be recalled that the tablet dissolution testing is one of the most important test during the developments of solid dosage forms (Ku et al. 2011).

The overall shapes of curves obtained at different temperatures are similar in both applied pressures (Figure 2).

Except for low temperature (291) for which the conductivity variation is negligible, this parameter dramatically increases during dissolution process, the time required to reach the equilibrium state corresponding to the complete disintegration of tablets, being the same (about 1800 s) for all experiments. The disintegration time value ranged in the interval (306-3780 s) communicated by Almukainzi et al. (2010) about dietary supplements-based tablets, knowing also that the USP disintegration standard for vitamin C tablets is of 1800 s max in water at 310 (Bhagavan & Wolkoff 1993). In this context, Brielles et al. (2008) has already showed that the kinetic monitoring by EC allows evaluating not only the time of complete dissolution of a tablet but also the nature of the disintegration process: Rapid disintegration, erosion regime and an intermediary regime. Nevertheless, the compression pressure appears to influence considerably the effect of temperature on ion migration. In fact, for compression pressure of 2000 Pa, the equilibrium EC at

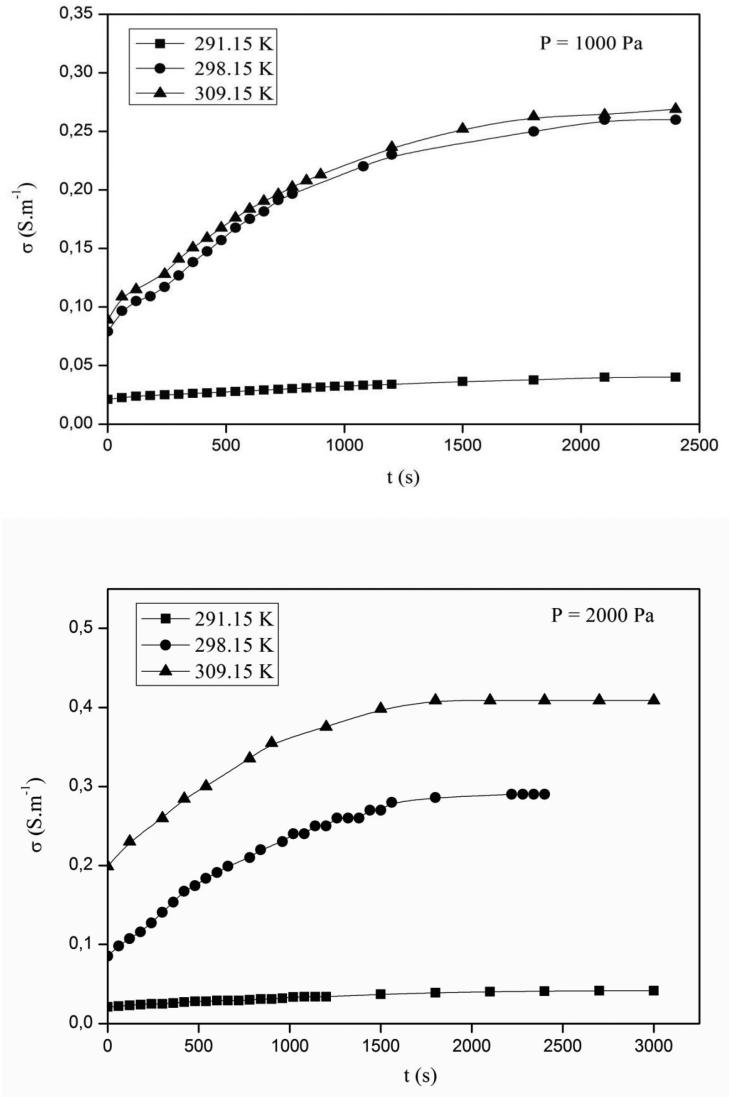

FIGURE 2. Conductivity versus time at different temperatures. Case of tablets from arbutus-berry powder obtained with a pressure of 1000 and 2000 Pa; surrounding liquid: bidistilled water

309 K is 1.5 times higher than that at 298, whereas for 1000 Pa, the curve shape for both temperatures are almost identical. This behavior could be linked to the complex mechanisms resulting from contact between tablets and surrounding liquid medium. This complexity is due to the physicochemical phenomena that occur when two solid phases (tablet) and liquid (surrounding liquid) are contacted: Penetration of certain molecules of the liquid in the interstitial spaces of the tablet; solubilization of organic substances (electrolytes and non-electrolytes); transport of solubilized molecules in the surrounding liquid; increase of the concentration of charged particles in the liquid phase which leads to an increase of the EC; interaction effects between (+) and (-) electric charges which can involve the complexation phenomena (case of electrostatic attraction) and therefore a decrease in conductivity; and delay in the disintegration process of agglomerates of fruit powder.

As that generally takes place, the tablet surface disintegrates into granules that in turn disaggregate into fine particles, this process being moreover intensified by the effect of the high temperature of 309 K. So, the heat effect can for example simultaneously accelerate the delitescence of the tablet surface and expulsion of the obstruent viscous liquid from capillaries and subsequently the ion transfer into liquid medium. These findings are supported by those communicated by Nilsson et al. (2003) who showed that the conductivity decreases with increasing tablet density.

TABLE 2. Parameters and statistical tests of the selected models related to dissolution of tablets obtained under pressures of 1000 and 2000 Pa, in distilled water

Pressure (Pa)	T (K)	Parameters and statistical test	Models		
			Peleg	Singh & Kulshrestha (1987)	Singh et al. (1981)
1000	291	R^2	0.893	0.987	0.966
		k_1 (m.s.S^{-1})	5.86×10^{-2}	-	-
		k_2 (m.S^{-1})	2.93×10^{-2}	-	-
		k (s^{-1})	5.00×10^{-4}*	1.25×10^{-3}	1.07×10^{-3}
		SEE	2.18×10^{-5}	3.21×10^{-5}	3.21×10^{-5}
		RMSE	4.67×10^{-3}	5.66×10^{-3}	0.66×10^{-3}
	298	R^2	0.939	0.956	0.987
		k_1(m.s.S^{-1})	44.37×10^{-4}	-	-
		k_2(m.S^{-1})	0.34×10^{-5}	-	-
		k (s^{-1})	7.68×10^{-4}*	1.59×10^{-4}	1.58×10^{-3}
		SEE	3.19×10^{-3}	3.34×10^{-4}	1.66×10^{-2}
		RMSE	5.65×10^{-2}	6.30×10^{-3}	1.29×10^{-2}
	309	R^2	0.960	0.952	0.9745
		k_1 (m.s.S^{-1})	4.18×10^{-3}	-	-
		k_2 (m.S^{-1})	0.36×10^{-5}	-	-
		k (s^{-1})	0.87×10^{-3}*	2.02×10^{-3}	1.77×10^{-3}
		SEE	1.87×10^{-3}	2.70×10^{-2}	3.05×10^{-2}
		RMSE	4.32×10^{-2}	1.64×10^{-1}	1.75×10^{-1}
2000	291	R^2	0.794	0.998	0.922
		k_1 (m.s.S^{-1})	54.50×10^{-3}	-	-
		k_2 (m.S^{-1})	2.97×10^{-5}	-	-
		k (s^{-1})	0.55×10^{-3}*	0.52×10^{-3}	1.38×10^{-3}
		SEE	4.81×10^{-5}	2.37×10^{-2}	8.26×10^{-4}
		RMSE	6.94×10^{-2}	1.54×10^{-1}	2.88×10^{-2}
	298	R^2	0.939	0.992	0.9261
		k_1 (m.s.S^{-1})	42.45×10^{-4}	-	-
		k_2 (m.S^{-1})	2.80×10^{-6}	-	-
		k (s^{-1})	0.65×10^{-3}*	0.50×10^{-3}	1.82×10^{-3}
		SEE	5.88×10^{-3}	5.54×10^{-1}	3.05×10^{-2}
		RMSE	7.67×10^{-2}	7.44×10^{-1}	1.75×10^{-1}
	309	R^2	0.974	0.9945	0.954
		k_1 (m.s.S^{-1})	87.87×10^{-6}	-	-
		k_2 (m.S^{-1})	3.40×10^{-6}	-	-
		k (s^{-1})	0.10×10^{-3}*	0.88×10^{-3}	1.87×10^{-3}
		SEE	1.00×10^{-1}	3.07×10^{-1}	2.72×10^{-2}
		RMSE	3.16×10^{-1}	7.85×10^{-1}	1.83×10^{-1}

* calculated as k_2/k_1

TABLE 3. Activation energy related to dissolution of tablets

Pressure (Pa)	Peleg		Singh & kulshrestha (1987)		Singh et al. (1981)	
	R^2	E_a (kJ.mol^{-1})	R^2	E_a (kJ.mol^{-1})	R^2	E_a (kJ.mol^{-1})
1000	0.833	21.380	0.886	18.740	0.824	19.248
2000	0.975	28.475	0.800	23.104	0.726	11.481

The average values of the parameters related to various mathematical models, as well as corresponding statistical data applied are recapitulated in Table 2. It must be noticed that the appropriate model is a priori unknown.

It must be precised that the k Peleg constant is calculated as k_2/k_1 ratio. As can be seen, whatever the temperature and compression force values, k_1 is higher than k_2, what is in conformity with the literature data devoted to the matter transfer (including water and other molecules) and implicating numerous food matrixes at different temperatures (Cox et al. 2012; Rhim et al. 2011; Yildirim et al. 2010). In addition, according to statistical data, the adequacy of models seems to be influenced by both parameters (temperature and compression force). However, the model of Singh and Kulshrestha appears to be more appropriate to describe the kinetics of dissolution of tablets, especially for compression force of 2000 Pa (R^2=0.99). On the contrary, concerning rehydration and dissolution of freeze-dried rice pulps, Rhim et al. (2011) have found that the model of Peleg fit more adequately experimental data, compared with that of Singh and Kulshrestha.

To the best of our knowledge, there are no data about the temperature dependence of ionic transfer in food matrixes-surrounding liquid systems, the values found regarding the activation energy (Table 3) are in concordance with that (20 kJ mole^{-1}) related to the diffusion of ions through liquids (Gronow 1987).

CONCLUSION

The results showed that Arbutus berry (*Arbutus unedo* L.) fruits can be easily processed into powder and then into tablets by direct compression. The obtained tablets are of various applications, namely their consumption as such and their use as a carrier for dried extract from other plant species.

In addition, the dissolution kinetics of the tablets, investigated throughout EC of the surrounding medium is before all correctly described by Sing and Kulshrestha model (R^2>0.99 in certain cases), whereas the activation energy ranged between 18.7 and 28.5 kJ.mol^{-1}.

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